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**DEVELOPMENT OF A FIELD-PORTABLE
OIL ANALYTICAL KIT**

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ABSTRACT

A portable kit has been developed specifically for measuring oil properties at the spill site. The kit is capable of measuring density, viscosity, flash point, water content and the chemical dispersibility of oil. This report describes the evaluation and selection of the methods and components used to collect, prepare, and analyze the oil. In addition, the report describes the measures taken to assemble the components into a portable kit. The evaluation of existing field tests is also reviewed.

RESUME

ACKNOWLEDGEMENTS

This project was funded by the Emergencies Science Division of Environment Canada, the United States Minerals Management Service, and the American Petroleum Institute. Mr. M. Fingas was the Scientific Authority. Illustrations in the Portable Field Kit Manual were drawn by Gerald W. Lavery.

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1.0 INTRODUCTION

The effectiveness of any countermeasure technique is to a certain degree dependent upon the physical characteristics of the spilled oil. Once oil is released into the environment, its properties are in a dynamic state due to weathering. This means that countermeasures must be adapted to deal with the changing characteristics of the oil. For example, the opportunity to use dispersants effectively is usually limited to the early stages of the spill before significant evaporation and emulsification have occurred. As well, the efficiencies of most mechanical recovery devices are limited by the rheological properties of the recovered material. Equipment that works well one day may become ineffective the next day because the viscosity of oil has increased beyond the capability of the equipment.

On-scene decision-makers must quickly assess the situation and then decide where and how to deploy the available resources. Usually the performance limits of equipment are known but not the properties of the spilled oil. At best, these properties can only be estimated. Sending samples away to be analyzed would be neither time effective nor practical. The utility of field analysis becomes evident.

The objective of this project was to develop a portable analytical kit which can be taken to the spill site and used to obtain rapid, reliable measurements of crucial oil properties. The kit would allow on-scene personnel to determine and monitor the properties of the oil. This information could then be readily incorporated into the operational decision-making process.

2.0 REQUIREMENTS OF THE KIT

The kit measures the following properties:

- Density

Oil density indicates the possibility of the oil sinking or being over-washed (Buist and Potter, 1987; Wilson et al., 1986).

- Viscosity

Viscosity is a measure of fluidity. Normally, it is the limiting factor for mechanical skimming and pumping equipment. Oil viscosity is also a major determinant of spreading.

- Dispersibility

This test will show if commercial dispersants are effective on the oil.

- Water content

The quantity of water in the sample indicates the extent of emulsification.

- Flash point

Flash point is a measurement of flammability, and provides an indication of potential fire hazards.

In order for specific equipment and methods to be considered for the kit, each had to meet the criteria outlined below:

- Equipment must be compatible with the limited space and weight requirements of a self-contained portable kit. The complete kit should be easily carried by no more than two persons, and can be shipped by common forms of transportation;
- Tests can safely be performed under the demanding operational conditions expected out in the field;
- Tests should be relatively simple to perform with little prior training and results must be easy to interpret;
- Results are to be within acceptable limits of accuracy and repeatability. Test procedures should be based upon generally accepted and standardized analytical methods;
- Definitive results are produced within hours of receipt of a sample.

3.0 EVALUATION OF EXISTING FIELD TESTS

3.1 Fina Oil Spill Test Kit

The Fina oil spill test kit was developed in the late 1970's by Labofina S.A.(Belgium) for the Dutch authorities Rijkswaterstaat. At the present time, it is the only portable kit available for measuring a variety of oil properties. A Fina kit was purchased and thoroughly tested.

Many of the test used in this kit are based upon empirical methods and not upon generally accepted analytical procedures. Measurements therefore usually require subjective interpretation by the operator. Consequently, the precision and accuracy depend upon the operator, the working environment, and the physical characteristics of the sample. Several tests involve difficult, time-consuming manipulations, and require relatively large amounts of oil. Little provision is made for cleaning the equipment, thus performing repeat measurements may be difficult. The kit and consumable supplies used for the tests are not readily available in North America.

There have been significant advances in analytical instrumentation since the development of the Fina kit. Modern instruments are more accurate, user friendly, and conform to standardized methods. Many of these instruments can be made field portable.

The Fina kit can measure 11 properties. The following is a summary of the evaluation of the tests of interest.

3.1.1 Specific Gravity

Specific gravity is determined using a modified spring balance and a conversion table. The balance consists of a plastic bottle attached to a spring. A graduated scale measures the difference in the vertical displacement of the spring resulting from the difference in weight of equal volumes of water and oil.

A table relates the values on the scale to specific gravity.

Two samples, mousse mix oil (see appendix B for composition) and the standard emulsion (see appendix A composition), were used to evaluate the method. Tests were conducted at room temperature. The spring balance gave specific gravity values of 0.90 to 0.95 for the mousse mix oil and 1 for the standard emulsion. Specific gravity values determined using an Anton Paar DMA35 density meter were 0.917 for the mousse mix oil and 0.985 for the standard emulsion. The spring balance was sensitive to motion. This made setting the scale to zero and reading values from the scale difficult. The bottle was difficult to properly fill with the viscous, semisolid emulsion. Emptying and cleaning the bottle between measurements was time-consuming. The test requires a large sample volume of 270mL.

3.1.2 Viscosity

Two methods are included in the Fina kit for measuring viscosity. A viscosity cup is used for low viscosity samples. This test requires 100mL of sample. The viscosity, in centistoke, is obtained by measuring the time required for the sample to flow out of a hole in the bottom of the cup. A table is used to convert time to viscosity.

Samples with high viscosities are measured by determining the diameter of an oil spot formed after a small sample of oil has fallen from a fixed height. The diameter is measured after a specified time using a scaled compass. Viscosity values in centipoise are read from the compass. Testing was performed at room temperature using the standard emulsion. Following the Fina procedure a value of 10,000cP was determined. Analysis of the emulsion using a Haake RV20 rotational viscometer indicates the emulsion is a non-Newtonian fluid. Viscosity is inversely proportional to the shear rate and ranged from 300,000cP to 2,000cP using a shear rate of 0s^{-1} to 100s^{-1} in 2 minutes.

When and how to determine the diameter of the oil spot was sometimes

difficult. The difficulty is due in part to the non-Newtonian nature of the emulsion which results in the mousse having inconsistent falling and spreading rates. Also, the spot formed was not always circular. Measurements require considerable operator judgement.

The major drawback of this method is that shear forces can not be controlled nor measured. The rheological properties of non-Newtonian samples can not be properly determined.

3.1.3 Dispersibility

The dispersibility of an oil is determined by shaking water, oil, and dispersant in a graduated cylinder for 10 seconds followed by a 30 second settling time. The volume of water-to-oil is 50:1, and the volume of oil-to-dispersant is 20:1. The water colour is then compared to a colour scale which allows an estimate of dispersant effectiveness.

An evaluation using the standard emulsion, a variety of crude oils, and various dispersants was carried out. Using the colour scale, dispersibility effectiveness values of about 90% were determined for the emulsion and less than 10% for the light coloured oils. A visual estimate of the amount of oil remaining on the surface of the water would indicate that these values were incorrect. The colour of the oil clearly had an effect upon the estimate derived from the colour scale. Estimates of effectiveness for dark coloured samples tend to be high while light coloured samples tend to give low estimates of effectiveness. Correlating test results to laboratory results was not possible because of the different settling times and oil-to-water ratio.

3.1.4 Water Content

A solvent extraction technique is used to determine the water content. A fixed volume of emulsion is placed in a customized flask and extracted with

a specialized solvent. After a settling period, a scale is used to measure the height of the water layer. A conversion factor is then applied to obtain a water percent content.

The limits of the method were evaluated using Norman Wells crude oil and the standard emulsion. The water content of Norman Wells crude oil measured by Karl Fischer titration was less than 1%. The Fina method was not capable of measuring this low of a water content. The water content of the standard emulsion measured by Karl Fischer titration was 70.32% by volume. Results using the Fina method ranged for 45% to 75% by volume. This large variance results from difficulties in measuring the height of the water layer due to incomplete separation of the water and solvent phases.

3.1.5 Flash Point

The flash point test is designed to provide a flash/no flash result for a closed cup test at 60°C. A crucible containing sample is placed into a sand bath maintained at 140°C to 150°C. A portable burner is used to heat the sand. After 90 seconds a flame is passed over the sample.

The apparatus has a high centre of gravity. This means it tends to be unstable, and could easily be knocked over if subjected to motion. If knocked over, the open flame and hot oil would be a potential danger. Inconsistent results were obtained from tests conducted at room temperature for the standard emulsion. Examination of the method showed that small differences in the depth of the thermometer lead to significantly different measurements. It was determined that a temperature gradient exists throughout the sand bath. How the sand was heated and placement of the thermometer affected the results. Measurements of samples assumed to be at 60°C, varied from 57°C to 85°C. Obtaining repeatable results required extreme diligence.

3.2 Field Dispersant Effectiveness Test Kit

A previous study by Ross (1988) examined four different field tests for determining dispersant effectiveness (Pelletier Screen Test, Fina Spill Test Kit, Mackay Simple Field Test, and EPA's Field Dispersant Effectiveness Test). All tests were designed to provide quick, qualitative results. The EPA's Field Dispersant Effectiveness Test (FDET) is commercially available (Sunshine Technology Corporation - West Hartford, Connecticut) and the Fina Dispersibility Test is part of the Fina Oil Spill Test Kit. The other test kits must be assembled by the user. Ross found that the portable tests, although simple to perform, had deficiencies. The most serious problem was the lack of correlation between the results of the field tests and accepted laboratory tests. Ross developed a test (the S.L. Ross Field Test) which overcame most of the deficiencies of the earlier field tests. It provided quantitative results of effectiveness which correlated with the Warren Spring Laboratory Rotating Flask Test.

4.0 EVALUATION OF EQUIPMENT FOR KIT

An extensive literature search of current methods and instruments used to analyze petroleum in the laboratory and in the field was carried out. Manufacturers were contacted and specific details regarding the equipment were obtained. Methods and apparatus that met the set criteria were selected for physical testing. Laboratory tests were conducted using a variety of oils and water-in-oil emulsions. Testing was conducted under conditions which simulated the operating conditions expected at a remote spill site or on a ship at sea. The sensitivity of tests to movement and to temperature were examined. Testing was performed at 15°C and 5°C. Procedures were condensed and simplified as much as possible for field application. Results obtained using the field procedures were compared with data from standard laboratory analyses.

Procedures were established for the five physical measurements of interest, as well as for collecting and preparing the oil samples.

4.1 Sample Collection

Equipment is provided in the kit to collect oil samples from both an oiled beach and from the water surface. Beached oil samples are collected using a spatula and stored in a Teflon container.

Collecting samples from a water surface may be difficult and time-consuming depending upon the condition of the oil and sea state. Several different types of sampling equipment are included, thus the procedure can be modified to suit the circumstances.

Common sample collectors such as bailers and dippers, proved difficult to operate and could not obtain sufficient quantities of sample. Previous studies (Daling, 1991; Seakem Oceanography Limited, 1990) have employed mesh baskets and nets to collect samples from water. Commercially available mesh baskets and nets such as the ones used to clean debris from swimming pools were evaluated. Viscous or emulsified oil samples are retained on the polyester net. However, due to the size of the mesh openings (approximately 2.5mm^2) less viscous oil samples passed through the net. To overcome this problem and to develop a more versatile sampler, different materials such as polyester, polypropylene, polyethylene and Teflon with mesh openings ranging from $21\mu\text{m}$ to $408\mu\text{m}$ were evaluated. The polyester mesh with a $105\mu\text{m}$ diameter opening was found to be the most effective, although no mesh was able to prevent very low viscosity oils from passing through. A sampling funnel was designed and developed for collecting very low viscosity samples. The funnel is made of Tedlar and equipped with a spout for draining off excess water. The nets and funnels were modified so they can be easily connected to a telescopic extension pole. A telescopic extension pole of the type used in the painting industry is provided.

4.2 Sample Preparation

Debris in samples, such as beach material and flora, can potentially damage the more sensitive instruments included in the kit, in addition to affecting the measurements. It is therefore important to remove any such interfering material from the samples. This must be done in such a way that there is minimal effect upon the properties of the sample.

Several different types of filtration apparatus were tested. These however were not able to filter viscous samples. A self-contained filter press is provided with the kit (Fann Model MB Filter Press, Baroid Testing Equipment - Houston, Texas). This filter press is extremely rugged and portable. It is designed to be used by the drilling industry for filtration tests of drilling mud. Carbon dioxide from small, disposable gas cartridges is used to provide pressure which forces the sample through a filter medium. The filter medium supplied by the manufacturer and used to test drilling mud consists of filter paper (Baroid or #50 Whatman) and a wire screen.

An evaluation of the filter press was performed using the standard emulsion and the manufacturer's filter medium. Two problems were noted: the filter medium broke the emulsion; and the flow rate through the filter medium was low. A new filter was developed that retained fine debris without breaking the emulsion while maintaining a good filtration rate. Tests to develop the new filter were conducted at room temperature using the standard emulsion mixed with sand (10% by weight) and various crude oils. The diameter of the sand particles ranged from 150 μm to 425 μm . Viscosities of the crude oils ranged from 1.140cP to 34,000cP at 15°C (Bobra and Callaghan, 1990). Different types of filters were evaluated with retention sizes ranging from 2.7 μm to 530 μm . The final design chosen was a filter paper with an 4cm hole cut in the middle over which a 105 μm mesh disk is placed. The filter paper is necessary in order to form a good seal within the apparatus. The 105 μm mesh disk provides a good flow rate and retains harmful particles.

4.3 Density

An Anton Paar DMA35 density meter (Anton Paar K.G. - Austria) is used to measure density. This hand-held instrument is battery-powered and provides digital readings in grams per millilitre ($\pm 0.001\text{g/mL}$) within seconds. It has an operational temperature range of 0 to 40°C and requires only 2mL of sample. The density meter uses the mechanical oscillator technique to determine density from a change in vibrational frequency. The procedure is similar to ASTM D4052-86 "Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter."

Other methods used to determine density or specific gravity were evaluated under simulated field conditions. These methods included the use of pycnometers, weighing bottles and hydrometers. It was concluded that these apparatus would be too difficult to use under field conditions. They tend to be sensitive to motion and difficult to use with viscous emulsions.

4.4 Viscosity

A review of the different methodologies used to determine viscosity was carried out. Laboratory testing was performed on those instruments which appeared to have potential for field use. The outcome of the tests indicated some of the limitations of particular methods. Capillary viscometers, which measure kinematic viscosity, are not well suited for measuring viscous non-Newtonian oils. Testing showed that falling needle/ball viscometers were not practical for measuring oil viscosity in the field. Measurements were affected by motion, and opaque samples can not be measured. Of the many different types of rotational viscometers available, few were suitable for the field kit. Some of the reasons include: their inability to measure absolute viscosity; their limited shear rate range; the need for conversion factors and calculations to determine shear rate and viscosity; and the requirement of a level and stable surface in

which to make the measurements.

A Bohlin Visco 88 BV viscometer (Bohlin Reologi Inc. - Sweden) was chosen for the kit. This variable speed rotational viscometer is fully portable and battery-powered. It can be operated as a hand-held instrument and provides a direct reading of viscosity in Pascal•seconds (1 Pascal•second = 1000 centipoise). Samples with viscosities from 0.006 to 350 Pa•s (6 to 350,000 cP) can be measured.

The Visco 88 provides many features that are normally found only in larger, more expensive laboratory viscometers. The viscometer has the capability to generate different types of rheological data. These can be used to characterize the non-Newtonian flow behaviour of samples, such as water-in-oil mousse. Direct readings from the instrument can be used to generate rheological flow curves (shear rate versus shear stress, and viscosity versus shear rate). The flow curves can then be used to calculate yield points and apparent viscosities. The viscometer can interface with a computer to increase the operating and data analysis capabilities.

4.5 Dispersibility

The use of dispersants remains an attractive countermeasure option for dealing with oil spills. At this time there are a variety of tests for measuring dispersant effectiveness. Unfortunately, different tests can yield very different values. It should be recognized that no test, not even an elaborate laboratory test, can fully simulate oceanographic conditions. Nevertheless, many recent advances have been made in understanding the variables that affect dispersant effectiveness. After reviewing the existing field tests (Pelletier Screen Test, Fina Spill Test Kit, Mackay Simple Field Test, EPA's Field Dispersant Effectiveness Test, and S.L. Ross Field Test), it was concluded that it was possible to draw upon all findings and develop a procedure that would avoid most of the artifacts and deficiencies of existing tests. A portable test will allow on-scene

personnel to examine the relative effectiveness of a dispersant on an actual sample of the spilled oil using indigenous water and the prevailing environmental temperature.

The portable test was designed in such a way as to allow the operator to make a quick qualitative observation of dispersant effectiveness, and to obtain a quantitative value of effectiveness. The difficulties associated with using visual methods were pointed out by Ross (1988) in his evaluation of field dispersant effectiveness tests. A major problem is that the colour of the oil affects the amount of oil perceived to be dispersed. If two oils of different colour were equally dispersed, the darker oil appears to be more dispersible. Therefore, assigning any kind of numerical value to effectiveness based on the appearance of the water containing the dispersed oil can be erroneous, if calibration standards are not prepared for comparison. These standards must use the same oil, water, and dispersant that will be used during the actual testing of the sample. A visual inspection will show qualitatively if the dispersant has had any effect. This can be done by comparing the results from a dispersant-treated oil against a non-treated oil. The non-treated oil will show an oil's natural dispersibility and thus act as a control. In order to obtain a valid measure of effectiveness, the amount of oil dispersed in the water must be analyzed using appropriate techniques.

Most techniques measure the amount of oil in water by extracting the oil from the water using solvents. Oil concentration in the extracted solvent is then determined analytically from calibration standards. Attempts were made to develop tests which could directly measure the amount of oil in water without using solvent extraction. A series of calibration standards were made by dispersing known quantities of oil in water. Different methods were tested to see if the turbidity of the dispersions could be correlated to oil concentration. Nephelometric methods of analysis were not capable of measuring the high turbidity values of most oil-in-water dispersions. It was also difficult to correlate nephelometric turbidity units (NTU) to oil concentration because the size,

shape, and refractive index of dispersed particles affect the light-scattering properties of the suspension. A spectrophotometer was used to determine the turbidity of oil-in-water dispersions by direct measurement of their absorbance. Stable dispersions could not be produced for calibration purposes. The inability to directly measure the oil content in water required that further method development use solvent extraction techniques.

The test method described here was chosen because it can be performed easily and rapidly, the results are relatively insensitive to minor variations in mixing energy, and the values obtained are repeatable and comparable to laboratory effectiveness tests. The test takes into account factors not considered by the previous field tests. Recent findings (Daling, 1988; Fingas et al., 1989; Fingas and Kolakowski, 1990; Nes, 1984) have shown the importance of certain variables on dispersant effectiveness testing. These variables are: the water-to-oil ratio; the length of settling time between the cessation of mixing energy and the withdrawal of a water sample; the extent to which the oil naturally disperses; and the manner in which the standards are prepared. It has been demonstrated that when the protocols of various existing laboratory tests are adjusted in such a way that these conditions are taken into account, the different tests yield comparable results (Fingas et al., 1989).

The procedure entails adding 200 μ L of oil (premixed with dispersant at a dispersant-to-oil ratio of 1:25 by volume) to 240mL of seawater contained in a 250mL Teflon separatory funnel. The funnel is hand-rotated at 30rpm for two minutes and then allowed to settle for 30 minutes. A 30mL water sample is drained into a 125mL separatory funnel where it is extracted with 15mL of dichloromethane. The same procedure is used for determining natural dispersibility, except that dispersant is not added. A set of standards is made up by adding 5 μ L, 15 μ L, and 25 μ L of oil (premixed with dispersant) to 30mL of water. The entire volume of each standard is then extracted using 15mL of solvent. The standards represent 20%, 60% and 100% dispersion respectively. Several solvents were evaluated on the basis solvency and hazardous

properties. Dichloromethane was chosen for use in the kit.

An estimate of the amount of oil that has been dispersed can be obtained by comparing the colour of the extracted solvent from the test runs (both the natural and chemical dispersibility) to the colours of the standards. An accurate determination of effectiveness can be made spectrophotometrically. The kit contains a hand-held, battery-powered Mini Spectronic 20 spectrophotometer (Milton-Roy Ltd. - Rochester, New York). The operator will use scaled graph paper to plot the transmittance values of the standards versus percent oil dispersed in order to obtain a calibration curve. The percentage of oil dispersed can be read directly from the graph.

4.6 Water Content

Different methods of breaking water-in-oil emulsions into distinct oil and water phases were tested. The techniques that were examined included distillation, centrifuge, thermal destruction and demulsifying agents. None of these techniques consistently broke stable mousse. Only solvent extraction techniques, in which a solvent breaks the emulsion by dissolving and extracting the oil, were capable of separating the oil and water into distinct phases. A variety of solvents were tested; including chlorinated solvents (chloroform, dichloromethane, trichloroethane, and Freon) and a less hazardous non-chlorinated solvent (toluene). It was found that chloroform and dichloromethane were the most successful at separating the oil and water. The volume of water is then used to calculate the water content of the mousse. Considerable operator skill is required for this procedure. Obtaining reproducible results can be difficult since the water content value must be interpreted visually.

A coulometric Karl Fischer titrator is designed to measure only very low concentrations of water (less than 1%). This method is not amendable for field use where samples contain high water content.

It was found that only the volumetric Karl Fischer titration technique

would analyze viscous emulsions reliably and consistently. The instrument that was chosen was a Metrohm 701 Karl Fischer Titrator (Metrohm Limited - Switzerland). This automated system can measure water content from 0 to 100%. Analysis takes only a few minutes and repeat measurements are easily performed. The instrument is self-cleaning and displays the calculated water content. The instrument has been equipped with a DC/AC inverter, thus allowing it to operate on either 120 volt AC or a gel cell (12 volt car battery).

The test procedure is analogous to the protocols for API MPMS (chapter 10.7), ASTM D4377-88 and IP 356/87 - "Standard Test Method for Water in Crude Oils (Karl Fischer) Titration." A 100 microlitre sample is injected into the titration vessel containing a solvent mixture (1:1:2, methanol:chloroform:toluene) which dissolves the emulsion. The free water is then automatically titrated to an electrometric end point with Karl Fischer reagent. The water content is displayed on the screen as a weight percent value.

4.7 Flash Point

The Setaflash Model 13740 (Stanhope/Seta - England) flash point tester was chosen for the kit. This same tester is included in a portable test kit used by the United States Navy to test naval fuel aboard a ship (Stavinoha et al., 1985). It is a portable unit powered either by a 120 volt AC source or a 12 volt DC, 4 amp battery. It has a measuring range of 0 to 100°C. The test is conducted as a flash/no flash procedure at two selected temperatures: the prevailing environmental temperature and 60°C. The procedure is based on ASTM D3828-87 and IP 303/80 "Standard Test Methods for Flash Point By Setaflash Closed Tester".

5.0 PREPARATION OF EQUIPMENT FOR FIELD USE

5.1 Modification of Equipment

A number of steps were taken to ensure the equipment included in the kit was capable of withstanding the conditions of field use. The labware included in the kit is either Teflon or polypropylene. Where possible fragile glass equipment parts, such as the bottles supplied with the titrator, were replaced with Teflon components. Peripheral equipment was selected on the basis of ease of use. All peripheral equipment such as syringes, stopwatches and micropipettes were tested to assess the effects of solvents, temperature, and motion.

Some of the equipment included in the kit are capable of doing more analysis than is required. Instrument parameters were streamlined and pre-set so that when the kit arrives at the spill site the equipment is in a ready-to-use state.

5.2 Transport Cases

For the kit to be safely shipped using common forms of transportation, the equipment and reagents had to be packaged in approved shipping containers and labelled according to appropriate regulations. Transportation regulations were reviewed and packaging consultants were contacted. The information and equipment necessary to meet current Transportation of Dangerous Good Act and International Air Transport Association rules were obtained and incorporated into the design of the kit.

Transport cases were designed and manufactured. The cases are fabricated from aluminum to protect the equipment during transport and in the field. The equipment is packed in foam and shipped assembled and ready-to-use. Peripheral supplies and equipment are arranged so that everything

required to perform a test is easily accessible.

The kit is contained in four cases. Each case is a self-contained "lab station", thus increasing the mobility of the kit. Case 1 contains the sampling equipment. Case 2 contains the solvents and equipment needed to remove debris from the sample and to perform the density, viscosity, and dispersibility tests. Case 3 contains a DC/AC inverter and the apparatus and solvents needed to determine water content. Case 4 contains the flash point tester and extra supplies needed for additional testing.

5.3 Manual

An illustrated manual has been compiled to provide detailed instructions. It contains: an inventory list of all the chemicals and equipment; a description of each test procedure; information for calibration and troubleshooting; and Material Safety Data Sheets.

Procedures are given as step-by-step instructions that will produce basic results. Additional information has been provided if more in depth analysis is desired. All appropriate data sheets and graphs are included. Calibration procedures are provided to check the accuracy of the equipment. A troubleshooting section covers some of the anticipated problems that may be encountered. Appropriate warnings and Material Safety Data Sheets are provided where needed. These were some of the measures taken to ensure the analysis can be performed safely.

6.0 RESULTS

The instruments and standard methods used for the laboratory analyses are listed in Table I. Table II illustrates that the results obtained for density, viscosity, flash point, and water content using the field kit instruments are in good agreement with measurements from standard laboratory methods. Figure 1 demonstrates the effect of filtration on the viscosity of the standard emulsion. Two samples of the standard emulsion were analyzed. For the standard emulsion, the effects of filtration were insignificant in comparison to the effects caused by other sources. These sources being the heterogeneity of the emulsion and the impact of shearing on the emulsion structure. It can be seen from Figure 2 that the Bohlin viscometer provides an accurate rheological characterization of non-Newtonian flow for the water-in-oil mousse.

Table I: Laboratory Equipment and Methods

Test	Method
Density - Anton Paar DMA 45 digital density meter	ASTM D4052 - 86
Viscosity - Haake RV20 rotational viscometer	DIN 53018
Flash Point - Pensky-Martens Closed Tester	ASTM D93 - 85
Water Content - Photovolt Coulometric Karl Fischer	ASTM D1533 - 88 Method B

Table II: Comparison of Test Kit Results and Laboratory Results

Test	Sample	Test Kit Result	Lab Result	Absolute Error
Density (g/mL)	ASMB* Crude	0.840(15°C)	0.8458(15°C)	0.0058
		0.847(0°C)	0.8514(0°C)	0.0044
	Norman Wells Crude	0.866(15°C)	0.8674(15°C)	0.0014
	Endicott Crude	0.915(15°C)	0.9154(15°C)	0.0004
	Emulsion	1.003(15°C)	1.0010(15°C)	0.0020
		1.005(0°C)	1.0041(0°C)	0.0009
Viscosity (Pa-s)	Standard	1.049	1.0007	0.0483
Flash Point (°C)	Jet Fuel A1	44	42***	2
	ASMB	6	7***	1
	Mousse Mix Oil**	72	71	1
Water Content (%)	Emulsion	69.31	69.00	0.31

* Alberta Sweet Mix Blend Crude Oil.

** this oil is a mixture of 50% Bunker C and 50% Alberta Sweet Mix Blend. The oil has been artificially weathered by air stripping; 7.7% by weight of the oil was evaporated off.

*** Data taken from Bobra and Callaghan (1990).

Figure 1: Viscosity versus Shear Rate Graph for Filtered and Non-Filtered Standard Emulsion

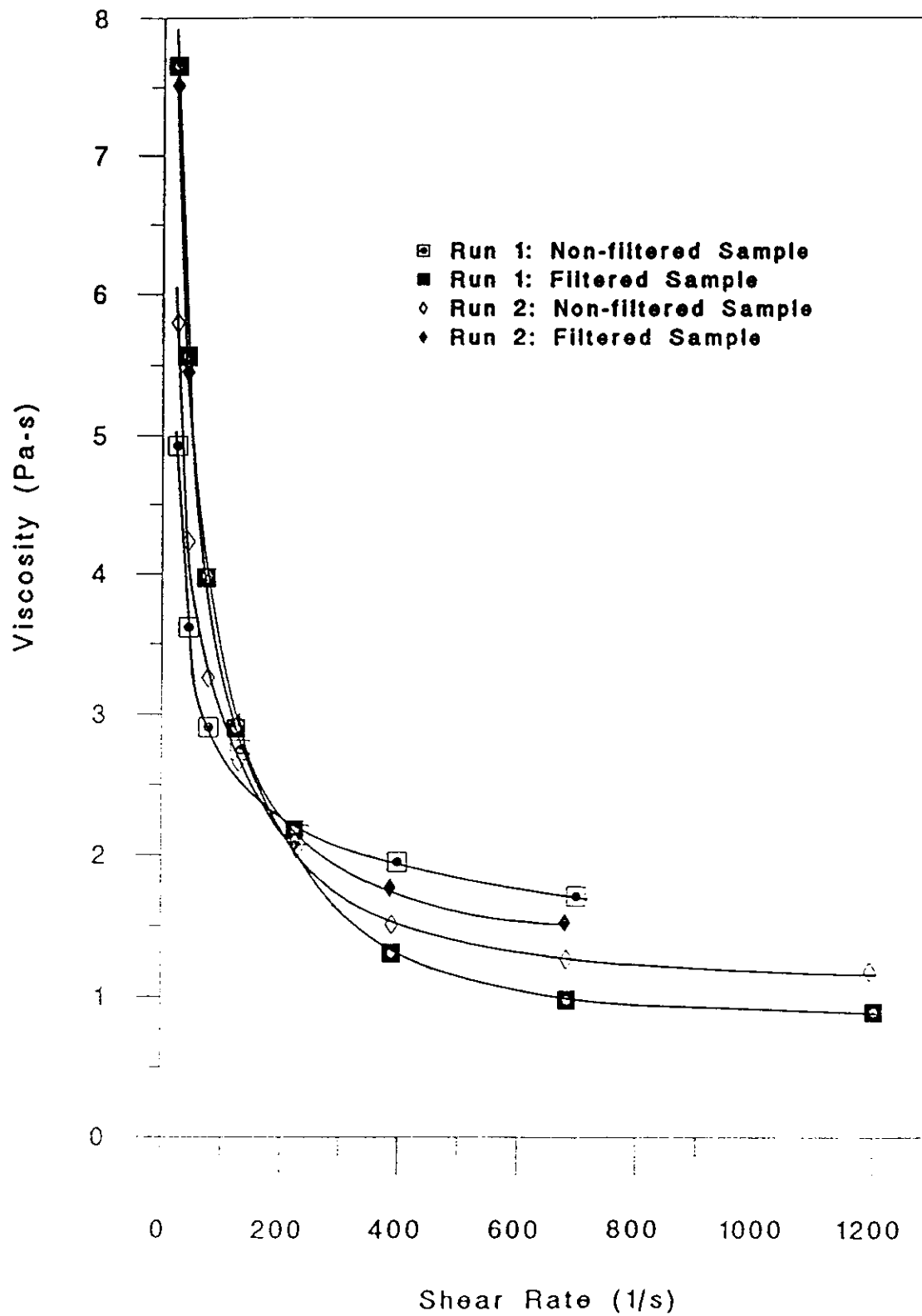
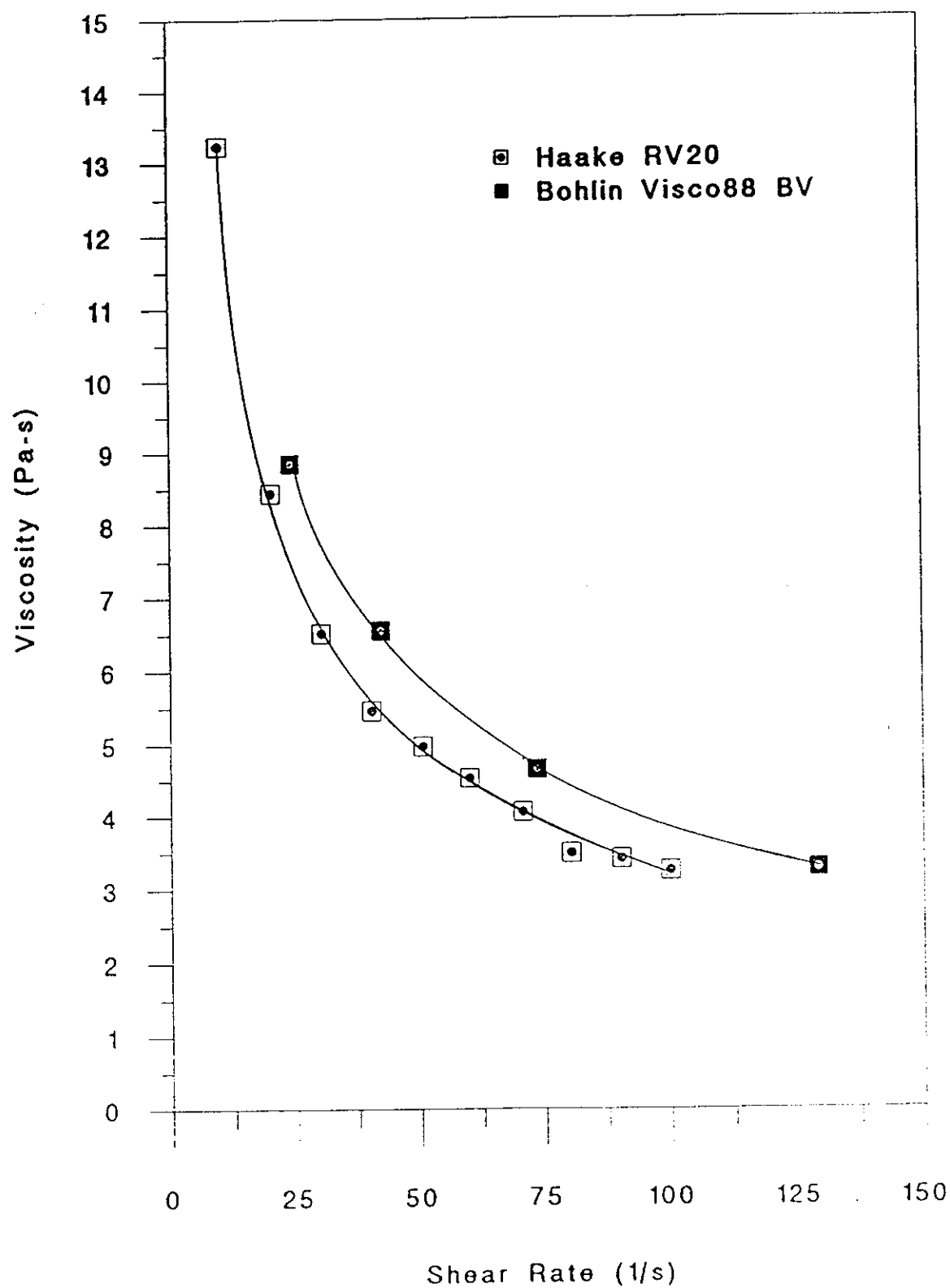


Figure 2: Viscosity versus Shear Rate Graph
Water-in-Oil Emulsion at 15 degrees Celsius



The dispersibility values for four oils as determined by the Portable Field Kit Test are presented in Table III, along with results obtained from the Warren Spring Laboratory Rotating Flask Test and the Swirling Flask Test. Procedures for the WSL Test and the Swirling Flask Test were taken from Martinelli (1984) and Fingas et al. (1989). Tests were conducted at room temperature using oil pre-mixed with Corexit 9527 at a dispersant-to-oil ratio of 1:25 by volume. For all tests, an oil-to-water volume ratio of 1:1200 was used, and the settling period was 30 minutes. All three tests rank the oils in the same order of dispersibility; Bunker C was the least dispersed and Alberta Sweet Mix Blend was the most dispersed. The results obtained using the Portable Field Kit Test are comparable with the other tests; the effectiveness values from the Portable Field Kit are in-between those of the Swirling Flask Test and those of the WSL Test.

Table III:

Dispersibility Results

Apparatus	Oil	Dispersibility %	No. of Data Points
WSL* Rotating Flask Test	Alberta Sweet Mix Blend	57% \pm 10%	9
	Norman Wells	54% \pm 4%	9
	Endicott	38% \pm 10%	9
	Bunker C	7% \pm 5%	9
Portable Field Kit Test**	Alberta Sweet Mix Blend	53% \pm 12%	52
	Norman Wells	31% \pm 10%	11
	Endicott	8% \pm 3%	13
	Bunker C	3% \pm 3%	5
Swirling Flask Test	Alberta Sweet Mix Blend	20% \pm 4%	12
	Norman Wells	20% \pm 2%	8
	Endicott	3% \pm 3%	8
	Bunker C	1% \pm 1%	8

* Warren Spring Laboratory

** data include runs where the rpm was varied slightly and the size of the separatory funnel was altered.

NOTE: All dispersibility results were measured after a 30 minute settling time.
The dispersibility results are written as the arithmetic mean plus/minus the standard deviation.

7.0 SUMMARY

Individual test components of a portable field kit were selected and tested in the laboratory under simulated field conditions. The methods were selected on the basis of portability, simplicity, safety, ruggedness, and reliability. A step-by-step manual has been compiled which provides detailed operation and calibration procedures as well as troubleshooting information and Material Safety Data Sheets. Cases have been designed and manufactured for transportation of the instruments and peripheral equipment. Besides protecting the kit during transportation, the cases also serve as "lab stations" for on-site testing.

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Appendix A - Standard Emulsion Composition

A standard emulsion was made by combining the reagents listed below in a high speed Warning blender and mixing (approximately five minutes) until emulsified.

1. 78% by volume artificial sea water: deionized water containing 3% salt by weight.
2. 19% by volume Mousse Mix oil: a mixture of 50% Bunker C and 50% Alberta Sweet Mix Blend. The oil has been artificially weather by air stripping; 7.7% by weight of the oil was evaporated off.
3. 3% by volume surfactant: Sorbitan Trioleate (Span 85) Lot# 15F-0173

Appendix B - Test Materials Used

Crude Oils

1. Mousse Mix oil: a mixture of 50 % Bunker C and 50 % Alberta Sweet Mix Blend by volume. The oil was artificially weathered by air stripping, 7.7 % by weight of the oil was evaporated off.
2. Alberta Sweet Mix Blend (ASMB) crude oil.
3. Endicott crude oil.
4. California crude oil - API gravity 11.
5. Panuk F-99 crude oil
6. Fuel oil #1 (Jet fuel A-1).
7. Norman Wells crude oil.
8. Prudhoe Bay crude oil.
9. Lube 27 lubricating oil.
10. Bunker C crude oil.
11. UDang crude oil.
12. North Slope crude oil.

Dispersants

1. Corexit 9527
2. Finasol OSR 2
3. Finasol OSR 5

Solvents

1. Dichloromethane
2. Chloroform
3. Methanol
4. Toluene
5. Trichlorotrifluoroethane
6. Trichloroethane

